Passive Fire Protection of Wood Substrates using Starch-based Formulations

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Introduction

Wood is the most sustainable, aesthetically pleasing and environmentally benign material. It often forms an integral part of structures and is the main source of furnishings found in homes, schools and offices around the world. Ligno-cellulosic materials such as wood also occupy a high-level hierarchy among natural materials, especially in the context of lightweight construction, which primarily derives from their near-carbon neutrality [1]. However, wood-based construction materials often suffer from a major drawback, i.e. its relatively high flammability. In addition, the toxicity and associated hazardous nature of fire resistant formulations that are currently in use for wood-based substrates are considered as serious drawbacks [2]. Therefore, it is highly prudent to devise means and methods of utilizing environmentally friendly, non-toxic and sustainable natural materials as passive fire protective agents for wood [3].
In the present paper, we report the preliminary results on the development, subsequent application and testing of some environmentally friendly formulations that are suitable for passive fire protection of ligno-cellulosic materials. The primary aim of this study was to evaluate the effectiveness of starch-based colloids as fire protective coatings of softwood substrates. The formulations were comprised of either an aqueous starch colloid solution alone, or in combination with some inorganic salts. The fire performance of unprotected wood and the wood samples with formulations applied as surface coatings was determined using a range of standard techniques. They included thermo-gravimetric analysis (TGA), Fourier Transform Infrared (FT-IR) spectroscopy, bomb and cone calorimetries, and a steady-state tube furnace-FT-IR.

Experimental

The planks of *Taeda Pine* softwood (ML Panel, UK) were conditioned for at least one week at 23±2 °C and 50±5% of relative humidity and then were cut to the required sizes. Potato starch powder, anhydrous sodium carbonate Na_2CO_3 and potassium carbonate K_2CO_3 were purchased from Fisher Scientific (UK), and diammonium hydrogen phosphate $(NH_4)_2HPO_4$ from Sigma Aldrich (UK). All chemicals were used as received. In all the experimental tests, an equal amount of starch-based colloid solution was applied uniformly onto the wood surface.
An IKA C200 bomb calorimeter (UK) was used to measure the gross calorific values according to the BS EN ISO 18125:2017. Thermo-gravimetric analyses (TGA) were carried out on *ca.* 5-10 mg samples, on a Mettler Toledo TGA/SDTA 851^e instrument in the temperature range of 30-800 °C, under both nitrogen and air atmospheres, at a heating rate of 10 °C/min. The flammability characteristics of the virgin and protected wood samples were evaluated by employing a bench-cone and standard cone calorimeters, at an external heat flux of 35 kW·m^{-2}. The wooden plaques of *ca.* 75 × 75 × 20 mm dimensions were used for the mass loss bench-cone calorimeter (Fire Testing Technology Ltd, UK) in accordance with the BS 476-13:1987 and ISO 13927 standards. The size of the samples for the oxygen consumption cone calorimeter (Dark Star Research Ltd, UK) was *ca.* 100 × 100 × 12 mm, and the measurements were carried out according to the ISO 5660 standard. The samples of softwood with

applied coatings had an average weight of 70 g and an average density of 0.61 g/cm^3. The analyses of gaseous products of wood thermal decomposition as a function of time were performed using a tube furnace-FT-IR technique at 350 °C and 650 °C. The FT-IR spectra were collected on a Bruker Tensor 27 spectrometer with OPUS Data Collection software.

Results

The values of the measured parameters pertaining to the combustion behaviours of the tested samples clearly demonstrated the overall efficacy of the starch-based coatings. This was evident from the increased times to ignition, increased char yields, reduced gross calorific values, lower mass loss rates of wood samples with applied coatings compared to the unprotected softwood. For example, in a bench-cone calorimeter, the coating based on colloidal starch solution with added K_2CO_3 demonstrated a delay of ignition by a factor of five compared to the starch treatment only, and by a factor of thirteen compared to the virgin wood material. In cone calorimetry, the protective coating containing K_2CO_3 made the ignition of the softwood extremely difficult to achieve. Moreover, this formulation, compared to the virgin material, reduced the net heat of combustion by 4 MJ/kg and also led to a significant drop of heat release rates (Table 1). Furthermore, the total smoke released was decreased by almost 50% when the softwood was protected with colloidal starch solution containing K_2CO_3. However, this sample produced a relatively high yield of carbon monoxide, which was also noted in the measurements using the tube furnace coupled to FT-IR. It was also observed that the coating based on colloidal starch formulation with $(NH_4)_2HPO_4$ demonstrated good char promoting attributes.

Table 1: Average values of flammability and heat transfer characteristics measured in the standard cone calorimeter.

Formulation	Ignition time (sec)	Time to flameout (sec)	pHRR (kW/m^2)	THR (MJ/m^2)	HRR (kW/m^2)	Net heat of combustion (MJ/kg)
None*	37.5	806.5	187.20	89.82	116.7	15.22
Starch	62.5	922.0	187.65	91.00	106.1	15.19
Starch + Na_2CO_3	137.0	976.0	136.20	64.64	76.1	12.09
Starch + K_2CO_3	191.5	1067.5	129.80	65.14	74.4	11.32
Starch + $(NH_4)_2HPO_4$	118.0	1123.50	148.00	87.14	86.4	14.46

* unprotected wood

In summary, the fire protective action of the starch-based coatings demonstrated a significant boost in the presence of the inorganic salts, and that K_2CO_3 seems to be the most promising among the additives tested in this work. The exact physio-chemical mechanism(s) underpinning this formulation cannot be identified with any certainity at present, but it is likely that such effects are initially triggered by the endothermic decomposition of the additive to produce carbon dioxide CO_2, which can in turn act as a diluent and a blowing agent for the char residues formed. These effects can also be attributed to the starch colloidal solution mixed with Na_2CO_3, albeit to a lesser extent, as reflected in the values of the corresponding empirical parameters. Given the ease of availability, low costs and non-toxic nature of these admixtures, they warrant further investigations with a view to exploring their potential use as protective coatings for ligno-cellulosic materials.

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